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US 4818509 A US 4591491 A US 4339419 A

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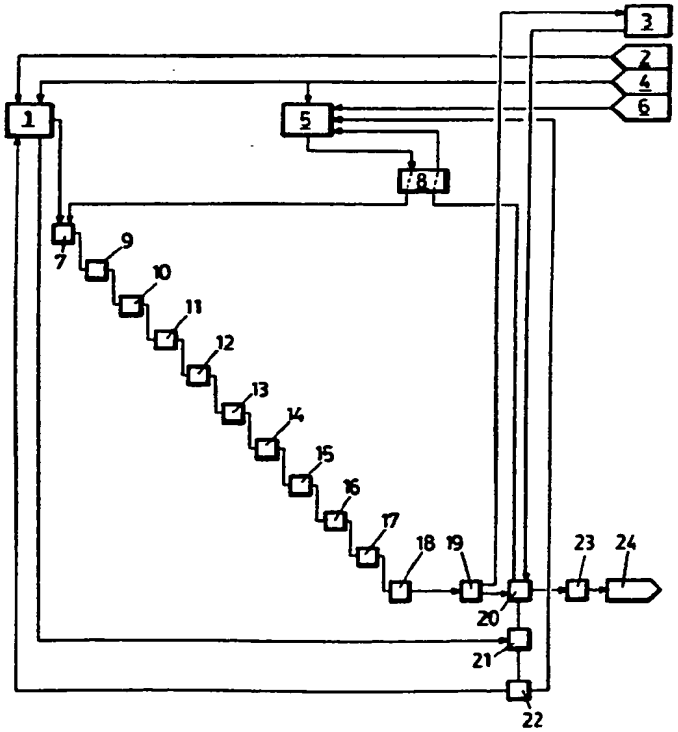
(54) Zeolite manufacture by a continuous gel crystallization process

(57) Zeolites are proposed by continuous crystallization of a gel through a cascade of crystallizers (preferably 10) 9 to 18, of increasing temperature throughout the series e.g. with an initial temperature of 80°C and a final temperature of 100°C, with controlled agitation and a maximum residence time from one crystallizer to the other of 5 minutes.

The gel is conventionally produced starting from reactors 1 and 5. In reactor 1 natural silica coming from tank 2 reacts with sodium hydroxide coming from tank 4, while in reactor 5 the aluminium mineral coming from tank 6 reacts with sodium hydroxide. The mass is cooled and concentrated in step 19 being afterwards filtered in 20, dried in 23 and final product Zeolite, is ready to be used in 24. Zeolite 4A for use in detergents is the preferred product.



At least one drawing originally filed was informal and the print reproduced here is taken from a later filed formal copy.

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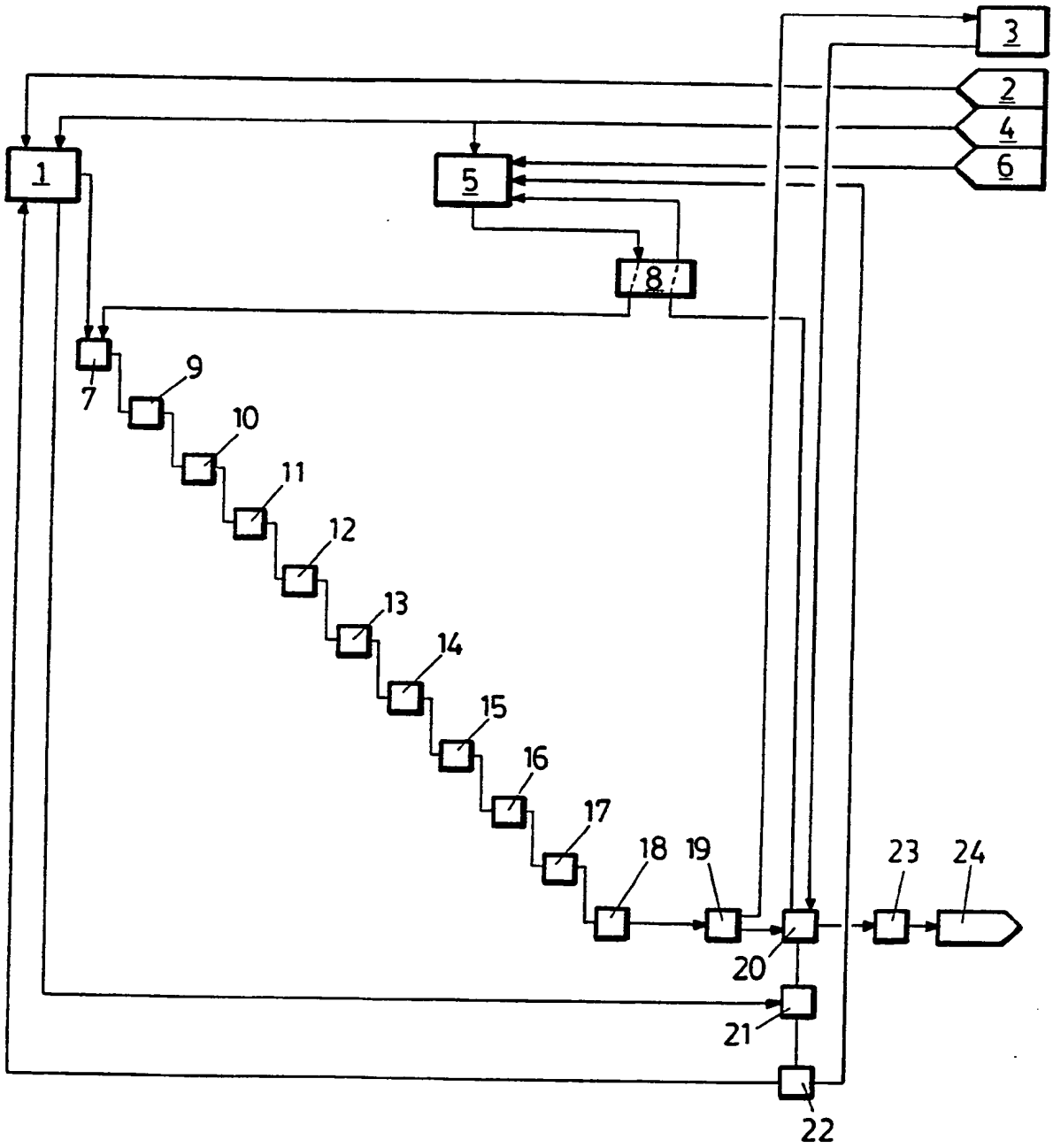

ZEOLITE MANUFACTURE BY A CONTINUOUS GEL CRYSTALLIZATION PROCESS

PURPOSE OF THE INVENTION

The invention is referred to a continuous gel crystallization process in order to manufacture zeolites to be used in detergents. This process has the characteristic of crystallization throughout a phase without any operative interruption, obtaining a product with a particle size between 1 and 7 microns, and an optimum calcium absortion capacity with less energy and saving in manpower compared with a repetitive cicle operative process.

This process requires less quality control compared with the traditional operation.

BACKGROUND OF THE INVENTION

Zeolite 4A is used in detergent manufacturing due to its capacity to retain the calcium present in the water.

However, it has been verified that the efficiency of Zeolite 4A in cloth washing does not only depends on Ca2+ absorption (washing water softening) but, basically, on the washing water Ca2+ depletion rate. The particle size of the product, completed in the crystalliztion phase, is decisive in relation to the kinetic reponse against of the Ca2+ in the washing process. Continuous crystallization, according to the present invention, allows a fine particle size adjutment and guarantees a regularity in the physical and chemical characteristics of the product.

STATE OF THE ART

It is not the first attempt of continuous crystallization. The document DE 2941636 proposed a multi-phase crystallization in a tubular reactor. This attempt has not achieved the desired results since the proposed crystallization is not flexible in

times, requires a continuous control and does not allow any particle size adjustment.

Against the prior art, the invention allows a stage by stage control in order to manufacture a final optimization.

High productivity and easy control are a need for any Zeolite crystallization process, and this is the purpose of this invention.

As an informative matter, we refer to patent documents with a non-continuous crystallization cicle, such as US 3310373 and BE 840315.

DESCRIPTION OF THE INVENTION

The process of this invention lies on passing the gel formed in a gellyfier by cascaded crystallizers (preferibly ten), each one provided with time and temperature controllers, with adjustable agitation, being the first crystallizier at 80°C of temperature, while the last one is at 100°C, with a temperature increasing in each crystallizer with a continuous renewal sothe reacting mass does not stay more than 5 minutes in each crytallizer.

DESCRIPTION OF THE DRAWINGS

In order to illustrate grafically what has been said, attached there is a drawing sheet representing a complete diagram of the Zeolite manufacture process including the crystallization process of this invention.

DESCRIPTION OF AN EXAMPLE OF REALIZATION

In reactor 1, natural silica from tank 1 reacts with sodium hydroxide from tank 4; in reactor 5 aluminum mineral from tank 6 reacts with sodium hydroxide from tank 4; the reaction product of reactor 1 passes to the gel preparation step 7,

while the reaction product of reactor 5 passes across the heat exchanger 8, and then, at 75°C of temperature, passes to the gel preparation step 7.

The gel from step 7 passes to the crytallizer 9 at 80°C of temperature, mantaining a controlled agitation and residence time. From crytallizer 9 it passes to crytallizer 10 continuing this way until it arrives to crytallizer 18, controlling time and temperature in each crytallizer, with a progressive temperature increasing until the achievement of 100°C and with a maximum temporary cicle of 5 minutes from one to the other.

The slurry obtained in the crystallization process is cooled and concentrated in 19, afterwards it is filtrated and washed in step 20, where the mother liquor is recicled after their passing by exchanger 8 to reactor 5. Washing waters pass to purifier 21 to be treated with an alkaline solution of SiO2. The resulting suspension is filtered in filter 22, being the liquid recirculated to reactor 1 and the solid to reactor 5. After drying in stage 23 at a temperature not above 110°C, Zeolite 4A is manufactured.

SUMMARY OF THE INVENTION

In brief, the invention lies on passing a gel coming from the jellyfying step 7 throughout several (preferably 10) cascaded crytallizers 9 to 18, with an initial temperature of 80°C in progression until a final temperature of 100°C, with controlled agitation and a maximum residence time from one crytallizer to the other of 5 minutes.

The gel is conventionally produced starting from reactors 1 and 5. In reactor 1 natural silica coming from tank 2 reacts with sodium hydroxide coming from tank 4, while in reactor 5 the aluminum mineral coming from tank 6 reacts with sodium hydroxide. The mass is cooled and concentrated in step 19, being afterwards filtered in 20, dried in 23 and the final product, Zeolite, is ready to be used in 24.

CLAIMS:

1. A process of preparing zeolite by continuous crystallization comprising preparing a gel by reacting together silicon dioxide (SiO_2) and aluminium oxide (Al_2O_3) in alkaline solution and passing the gel through a cascade of crystallizers, the temperature in each crystallizer being controlled such that in each crystallizer (except the first) the temperature is greater than the temperature in the immediately preceding crystallizer.

2. A process according to claim 1, wherein there are ten crystallizers in the cascade.

3. A process according to either preceding claim. wherein the level of the gel in each crystallizer is controlled.

4. A process according to any preceding claim, wherein the gel is subjected to controlled shaking in each crystallizer.

5. A process according to any preceding claim, wherein the temperatures in the crystallizer vary from 80°C to 100°C.

6. A process according to any preceding claim, wherein residence time of the gel does not exceed five minutes.

7. A process according to any preceding claim, wherein the zeolite produced is washed with water and the washing water recycled for use in preparing the gel.

8. A process according to any preceding claim wherein the zeolite produced is subjected to one or more operations selected from cooling, concentrating, filtering and drying.

9. Gel continuous crystallization process in order to manufacture Zeolite characterized because it lies on passing a gel conventionally formed in a gel reactor starting from the reaction

of two alkaline solution of SiO2 and Al203, by a plurality (preferably ten) cascaded crystallizers, each one provided with level control and adjustable agitation, being the first crystallizer at a temperature of 80°C while the last one is at 100°C, producing a temperature increasing between consecutive crystallizers, passing from one crystallizer to other assuring a total drainage in a maximum of 5 minutes, and being afterwards cooled, concentrated, filtrated and dried according to any conventional operation.

10. A process of preparing zeolite, substantially as described herein with reference to the accompanying drawings.

Patents Act 1977
Examiner's report to the Comptroller under Section 17 (The Search Report)

Application number
9102056.0

Relevant Technical fields

(i) UK Cl (Edition K) C1A (ACE)

(ii) Int Cl (Edition 5) C01B

Search Examiner
G A CLARKE

Databases (see over)

(i) UK Patent Office

(ii) ONLINE DATABASES: WPI, CLAIMS

Date of Search
11 March 1991

Documents considered relevant following a search in respect of claims
1 to 10

Category (see over)	Identity of document and relevant passages	Relevant to claim(s)
X	US 4818509 (MOBIL) See whole document	1 at least
X	US 4591491 (HENKEL) See Claim 1 and Figures	1 at least
X	US 4339419 (PEGUSSA; HEIKEL) See Claim 1	1 at least

Category	Identity of document and relevant passages	Relevant to claim(s)

Categories of documents

X: Document indicating lack of novelty or of inventive step.

Y: Document indicating lack of inventive step if combined with one or more other documents of the same category.

A: Document indicating technological background and/or state of the art.

P: Document published on or after the declared priority date but before the filing date of the present application.

E: Patent document published on or after, but with priority date earlier than, the filing date of the present application.

&: Member of the same patent family, corresponding document.

Databases: The UK Patent Office database comprises classified collections of GB, EP, WO and US patent specifications as outlined periodically in the Official Journal (Patents). The on-line databases considered for search are also listed periodically in the Official Journal (Patents).

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